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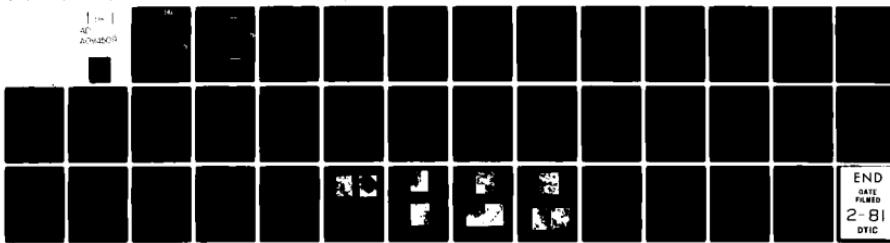
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A FUNDAMENTAL STUDY OF THE PROCESSING OF OXIDE DISPERSION STRENGTH--ETC(U)  
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A FUNDAMENTAL STUDY OF THE PROCESSING OF OXIDE  
DISPERSION STRENGTHENED METALS

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## I. INTRODUCTION

During the past four years we have been engaged in research on the processing of oxide dispersion strengthened (ODS) metals. We have conducted research on the fundamental variables of mechanical alloying and used this knowledge to synthesize a variety of different kinds of alloys. The main thrust of our work has been on the development of dispersion strengthened aluminum alloys by mechanical alloying. We have developed techniques for producing fine oxide and carbide dispersions in aluminum by mechanical alloying and have produced test material from powders using vacuum hot pressing and hydrostatic extrusion. We have studied the structure of these materials using transmission electron microscopy and have shown that the oxide and carbide dispersoids which form during mechanical alloying are amorphous in form and become crystalline only after subsequent heating. We have also studied the mechanical properties of these materials and found that they exhibit excellent strengths at elevated temperatures. In addition we have studied the deformation processing of ODS Ni base alloys and have found that germinative grain growth, which is so important in these materials, is most likely initiated by the growth of embryos found in as-hot worked structures.

In Section II of this Report, the principal findings of our research are briefly reviewed. This work is described in more detail in the previous Interim Scientific Reports and in the papers which have been and are being published. The work completed during the final year of this project is described in more detail in Section III of this Report.

## II. SUMMARY OF RESEARCH ACCOMPLISHMENTS DURING THE PAST FOUR YEARS (1976-80)

The purpose of this research has been to develop a better understanding of the synthesis and processing of ODS alloys. This work was motivated by the knowledge that the properties of ODS alloys depend very sensitively on processing, which in turn is very poorly understood. Most of what is known about processing does not appear in the open literature, with the consequence that attempts to make and use these interesting materials is often met with failure.

Two areas of research were identified for fundamental study: Mechanical Alloying and Deformation Processing. Since the development of mechanical alloying by Benjamin about 10 years ago, no basic university research had been done on the process. Our work is still the only academic work in the United States along these lines. In the field of deformation processing a great amount of industrial research has been done, but most of this has been directed toward the development of specific ODS alloys rather than toward the elucidation of fundamental mechanisms associated with processing. Our work has been focused mainly on mechanism of germinative grain growth and the structural cause of this process.

### Mechanical Alloying

We have made a number of studies of the basic stages of the mechanical alloying process, including cold welding, fracturing and the balance between these to produce steady state processing. We have found that the steady state processing can be characterized by equiaxed powder particles, a constant particle size distribution and a saturation hardness. Our work has shown the importance of the lubricant in controlling the balance between cold welding

and fracturing. This was known prior to our work but its importance had not been emphasized in the open literature. Indeed, we have shown that mechanical alloying cannot be accomplished if cold welding is not inhibited.

One of the remarkable features of mechanical alloying is that extremely fine mechanical mixtures can be produced in a very short period of time. Indeed the layer dimensions within mechanically alloyed (MA) powders can approach atomic dimensions after a short while in a high speed shaker mill. We have shown how this results from the geometric multiplication of layers when powder particles are continually fractured and re-welded to each other.

We have found that the organic lubricant used in the mechanical alloying of aluminum has two functions. The first is to facilitate mechanical alloying as discussed above, and the second is to supply carbon to the material for the formation of aluminum carbide. We have shown that aluminum carbide particles make a substantial contribution to the strength of MA aluminum alloys.

Our research has shown that cold welding during mechanical alloying can be delayed by lowering the milling temperature. We are not aware of any other reports of this effect. This finding is important because it provides a technique to accomplish mechanical alloying without adding extraneous organic material. We have used this technique to make a number of different materials by mechanical alloying.

In the course of our study of mechanical alloying of aluminum it became apparent that the technique could be used to synthesize other materials. In a related research project we have used the mechanical alloying process to make fine mechanical mixtures of Nb and Sn in powder form. When these powders

are combined with Cu powders and consolidated into wire by hydrostatic extrusion or wire drawing, and then heated to a high temperature, a superconducting wire is formed. We have also used the mechanical alloying process to make Ni-Al alloys and to create very fine dispersions of  $Y_2O_3$  within the microstructure of Ti-6Al-4V. The purpose of this latter work was to use  $Y_2O_3$  to stabilize the grain size of the superplastic titanium alloy.

Development of a New Kind of Dispersion Strengthened Aluminum Alloy by Mechanical Alloying

One of the principal objectives of this research program has been to use mechanical alloying to develop a new kind of dispersion strengthened aluminum. Based on our knowledge of sintered aluminum powder (SAP) materials, we reasoned that mechanical alloying of aluminum would result in the formation of an extremely fine dispersion of oxide within the metal. We assumed that this would occur naturally as the freshly exposed surfaces of the powder particles oxidize and become embedded into the powder by cold welding.

Our work has shown that very uniform dispersions of particles are formed in aluminum by mechanical alloying. As noted above, some of these particles are aluminum carbides when organic lubricants are used in the processing. The size and spacing of these particles is almost on the same scale as the precipitates which form in precipitation hardened alloys. As a consequence, the yield strengths of these materials are quite high. However, the most impressive mechanical properties are found at high temperatures. The dispersion strengthened aluminum we have made is considerably stronger than SAP, even though our material contains a much smaller amount of the dispersoid phase. We have characterized the impressive high temperature mechanical properties of this material in terms of a threshold stress for flow which

depends quite strongly on temperature.

In the course of our work on dispersion strengthening of aluminum by mechanical alloying we became aware of Benjamin's work along similar lines. Since that time INCO has developed at least one new commercial alloy (IN 9051) based on the mechanical alloying process.

Prior to our work the phases responsible for the strengthening of MA aluminum were not known with certainty. It was assumed that  $\text{Al}_2\text{O}_3$  would be present and carbon was known to have a strengthening effect. We conducted the first detailed TEM study of the formation of dispersoid phases in MA aluminum. We showed that the phases present in the mechanically alloyed state are amorphous in form and become crystalline only after heating. The temperatures required for ordinary consolidation (hot pressing or hot extrusion) are sufficient to cause the particles to crystallize. We have shown that both  $\gamma\text{-Al}_2\text{O}_3$  and  $\text{Al}_4\text{C}_3$  are present in MA aluminum (processed with an organic lubricant) after heating.

#### Origin of Elongated Grains in Recrystallized ODS Alloys

A considerable amount of effort has gone into the study of recrystallization of ODS alloys. Our own approach to this has been to use TEM techniques in an attempt to discover the origin of elongated grains. During the past year our study of the structure of as hot-worked MA 753 has given an important clue to this problem. We have observed embryos in the hot-worked material which have the same shape and orientation as the large grains which form after high temperature annealing. We believe this will allow us to give, for the first time, a mechanistic description of the origin of elongated grains in ODS alloys. More details on this work are given in Section III of this Report.

### III. RESEARCH REPORT FOR THE PREVIOUS YEAR (1979-80)

In this Section of the Report we present a more detailed discussion of some of the research results obtained during the past year. The work described here reflects the shift in emphasis in our work from the synthesis of ODS alloys to the study of their structure and mechanical properties. This change in direction brings to a close our work on the synthesis of ODS alloys. At present, all of our work is being conducted on commercially produced materials.

We have conducted a study of the high temperature deformation properties of IN-9051, a dispersion strengthened Al-Mg alloy now available for use in aircraft structures. Our attention has been focused on the interactive effects of dispersion strengthening and solid solution strengthening of this alloy. Our studies of the compression flow properties of this alloy indicate a variety of unusual effects (negative strain rate sensitivities, serrated flow, threshold stresses) which need to be understood. During the past year we have developed a model of plastic flow for this material which allows us to explain some of the unusual properties. In particular, we have shown that dislocations should lose their solute atmospheres when they pass particles by the Orowan mechanism. This is caused by the very high dislocation curvature forces that occur during the Orowan passing process. This mechanism helps to explain why dispersion strengthened Al-Mg alloys are weaker than Al-Mg solid solutions above the Orowan stress.

We have long been interested in the mechanisms by which large elongated grains form in ODS superalloys. During the past year we have used Transmission

Electron Microscopy to study the grain structure in as-hot worked and annealed MA 753. We believe we have observed the embryos that are responsible for germinative grain growth. Very small needle shaped grains aligned parallel to the working direction and having as <002> crystallographic axis have been found in as-hot extruded material. These small grains have the same crystallographic orientation and shape as the large elongated grains that form on annealing. Further work along these lines is expected to lead to a direct observation of the directional recrystallization process.

Our recent work has suggested that diffusional creep may be of some importance in creep and failure of ODS alloys. During the past year we have developed a theory for diffusional creep that applies to materials having elongated grains. The theory is general in that it considers both lattice diffusion and grain boundary diffusion controlled processes and applies to solids with grains of any length, width or thickness. This theory should be of use to us in our future work on diffusional flow effects in ODS alloys.

A. A Study of Dispersion Strengthening in Class I Solid  
Solution Alloys

(W. C. Oliver)

Virtually all research that has been done to date on dispersion strengthened metals has dealt with materials for which the major phase is, or behaves like, a pure metal. A common approach to understanding the creep properties of such dispersion strengthened "pure metals" is to assume that the particles give rise to a threshold stress, below which creep essentially does not occur. While this description has the virtue of simplicity and is reasonably accurate, it is difficult to imagine that the particles could have such a simple effect on the strain hardening and recovery processes that occur during pure metal-type creep. As a consequence, the origin of the threshold stress in creep of dispersion strengthened metals remains unexplained.

Although the creep properties of dispersion strengthened pure metals are obviously of interest, in any commercial application, the dispersion strengthening mechanism will be combined with other high temperature strengthening mechanisms. In particular, some form of solid solution strengthening would be a natural feature of any dispersion strengthened alloy.

Because the creep properties of Al - 3% Mg (a solid solution alloy) are reasonably well understood in terms of solute drag effects, it seems worthwhile to study the effects of dispersion strengthening in this regime. We have studied the creep properties of Al - 3% Mg which contains a dispersion due to mechanical alloying. This material (designated IN-9051) has been provided by the International Nickel Company. This material is expected to contain MgO particles and possibly particles of  $\text{Al}_2\text{O}_3$  and  $\text{Al}_4\text{C}_3$ . Figure 1

shows the temperature compensated strain rate as a function of the modulus compensated steady state flow stress for IN-9051. These data are the results to date of elevated temperature constant crosshead speed compression tests. A Hewlett-Packard 9825 data acquisition system in conjunction with an Instron testing machine was used for these tests.

There are two stress regions depicted in Figure 1 (designated as A and B). In each region the behavior of the material is somewhat different. In the following discussion it will be convenient to describe the steady state flow behavior with an equation of the type

$$\frac{\dot{\epsilon}}{D} = A \left(\frac{\sigma}{E}\right)^n$$

In region A, at high stresses, the stress exponent  $n$  is about 5 at lower stresses and increases at higher stress. The data in this region also shows that at a given stress, the material with a dispersion appears to deform faster than the matrix material without a dispersion (solid curve). This suggests that the material is weakened by the addition of the dispersion. It is interesting to note that the dispersion strengthened material appears to follow an extension of the climb controlled portion (dashed line, Fig. 2) of the matrix material curve. This indicates that the presence of the dispersoid effectively circumvents the micro-creep glide controlled creep mechanism. The temperature dependence of the strain rate is also the same as that for the matrix material; that is, the temperature dependence of lattice diffusion. It is also important to note that power law breakdown occurs at approximately the same stress in both materials, indicating that the low temperature strengthening due to the solute is unaffected by the presence of the dispersoid.

At the boundary between regions A and B in Fig. 1, the material appears to exhibit a very high, negative stress exponent. However in the strain rate

regime where the negative stress exponent appears, the material deforms inhomogeneously by the formation of one or several slip bands. This plastic instability is an indication of a very high stress exponent. However, because the deformation is inhomogeneous the small stress differences that result in a negative stress exponent are subject to question. The conclusions that can be safely drawn from the data are that at the boundary between the two stress regions the stress exponent is very large and that the position of the boundary is a function of temperature.

The stress exponent in region B of Fig. 1 is high and again becomes very high near the boundary between the two regions. One also observes an increased temperature dependence in this region. The high stress exponent is indicative of the effect of a dispersoid on the creep properties of the material.

From this preliminary work it can be seen that some characteristics of each of the strengthening mechanisms are observed when they operate simultaneously. It is evident that their interaction is interesting, but cannot be explained in a simple manner.

An important question to be answered is how the presence of the dispersoid can, under certain conditions eliminate diffusion controlled glide as a rate limiting mechanism. To determine how this might occur one must consider how a solute saturated dislocation will react when it encounters dispersoid particles during glide.

Figure 2 shows the geometry of a dislocation line that has just passed a particle. If the stress is high enough the particle will be passed by the Orowan bowing and the angle  $\theta_3$  will be 0. However, below the Orowan stress this will not occur and the dislocation must climb over the particle. In this case  $\theta_3 > 0$ . In any case Fig. 2 is general for a dislocation having just

passed a particle.

Due to the line tension of the dislocation the force on the dislocation at a given point may be directly related to the curvature of the line. For this reason one can see that there is an extremely high force on the dislocation at point A in Fig. 2. If the dislocation has a solute atmosphere it will certainly be separated from it at this point. The portion of the dislocation that breaks free from its atmosphere will move ahead with a much greater velocity than the saturated portion. This, in turn, can cause the entire dislocation to separate from its atmosphere by un-zipping. This un-zipping will occur only if the energy (E) of the entire dislocation line decreases with an increase in the un-zipped length. That is, un-zipping will occur when

$$\frac{dE}{dl} < 0 . \quad (1)$$

To determine if this condition is met one must write an expression for the total energy of the system as a function of un-zipped length and differentiate that expression with respect to the un-zipped length (or some parameter that varies directly with the un-zipped length). We will differentiate with respect to the angle  $\theta_1$ . (See Figure 1).

The expression for the total energy for a given value of  $\theta_1$  is

$$E = r \theta_2 \Gamma_0 - 2 R \theta_1 \Gamma - A \tau b \quad (2)$$

where  $\Gamma_0$  = the dislocation line energy without a solute cloud

$\Gamma$  = the dislocation line energy with a solute cloud,  
and,  $\tau$  = applied shear stress.

If we differentiate with respect to  $\theta_1$  we obtain

$$\frac{dE}{d\theta_1} = r \Gamma_0 \frac{d\theta_2}{d\theta_1} - 2 R \Gamma - \tau b \frac{dA}{d\theta_1} . \quad (3)$$

For the two line energies, we will use expressions given by Friedel [1]

$$\Gamma_0 = \frac{\mu b^2}{2} \quad (4)$$

$$\Gamma = \Gamma_0 - c_1 |w_M| r_1 / b^2 \quad (5)$$

where  $c_1$  = concentration of solute at the dislocation and is given by [2]

$$c_1 = \frac{1}{1 + \frac{(1-c_0)}{c_0} \exp [w_M/kT]}$$

$w_M$  = The binding energy between the dislocation and the solute atom

$r_1$  = The radius of the solute cloud ( $r_1 \approx b$ )

$c_0$  = Bulk solute concentration

$k$  = Boltzmann's constant

$T$  = Temperature.

The radii which appear in Eq. (3) are given by

$$R = \frac{\Gamma}{\tau b} \quad (6)$$

$$r = \frac{\Gamma_0}{\tau b} \quad . \quad (7)$$

From the geometry shown in Fig. 2, the following relationships can be obtained.

$$\frac{d\theta_2}{d\theta_1} = 2 \frac{R \sin (\theta_1 + \theta_3)}{r \cos \theta_2/2} \quad (8)$$

$$\frac{d\Lambda}{d\theta_1} = Rr [(\sin \frac{\theta_2}{2}) (\cos \theta_1 + 2 \tan \frac{\theta_2}{2} \sin (\theta_1 + \theta_3))] + \quad (9)$$

$$+ R^2 [\sin (\theta_1 + \theta_3) (\sin (\theta_1 + \theta_3) - \sin \theta_3) + \cos \theta_1 - 1]$$

Substituting Eqs. (4) - (9) into Eq. (3) yields a solution for the change in energy for a change in  $\theta_1$ . Figure 3 is a plot of the results of this calculation for a variety of applied stresses (expressed as percentage of the Orowan stress). One can see that for stresses above approximately 60% of the Orowan stress Eq. (1) is satisfied for all values of  $\theta_1$ . Therefore, at this stress or at higher stresses the dislocation will be completely separated from the solute atmosphere. Figure 4 shows the lowest stress for which the un-zipping condition (Eq. (1)) is met as a function of temperature. For all temperatures this stress is less than the Orowan stress.

One can see from these results that each time a dislocation passes a particle by the Orowan mechanism it is stripped of any solute atmosphere it might have. A conclusion that may be drawn from these calculations is that above the Orowan stress the presence of the dispersoid particles does not allow the maintenance of a solute atmosphere. For this reason diffusion controlled glide cannot be the rate limiting process at stresses above the Orowan stress in a material that is both solute and dispersion strengthened. This result explains the observed behavior of the material in region A of Figure 1.

We are presently trying to model the behavior of this material in the low stress region. Below the Orowan stress the particle passing mechanism must be one of climb of the dislocation over the particle. The model being developed will consider the interaction of a climb process with solute drag controlled glide.

B. A Study of the Mechanism of Germinated Grain Growth in MA 753

(J. K. Gregory)

The purpose of this study is to gain an understanding of the fundamental mechanisms controlling abnormal grain growth in MA 753. This alloy, like others in its class is a nickel-base superalloy but is distinguished by the addition of a finely dispersed oxide phase (Ni-20 Cr-2.4 Ti - 1.5 Al - 0.07 Zr-0.007 B-1.2  $Y_2O_3$ ). Consolidation of this alloy is accomplished by the conventional powder metallurgy processes of compaction and sintering followed by extrusion or hot-rolling. Both extrusion and rolling impart some directionality to the structure of oxide dispersion strengthened (ODS) nickel alloys. Subsequent annealing then produces a coarse elongated grain structure having desirable mechanical properties. It is hoped that an understanding of how this occurs can be used to improve and extend the effects of this type of processing.

The experiments performed involved both optical microscopy and transmission electron microscopy (TEM) to correlate the structure observed at high magnifications with the working direction. An as-hot extruded and hot-rolled bar of MA 753 was cut to produce slices approximately 1.5 mm (1/16") thick. Two orientations were chosen. One perpendicular to the extruding direction, and the other perpendicular to the short transverse direction. These pieces were then examined in the as-received condition and after annealing in air at 1315°C (2400°F) for ten minutes. TEM specimens were prepared by mechanically grinding the pieces to 250  $\mu m$  (10 mil) thickness. In the case of the specimens cut perpendicular to the short transverse direction, 600 grit surface scratches were made parallel to the extruding/rolling direction. Discs were cut and then electropolished in a 20% perchloric acid 80% ethanol solution at 20 volts. The

electropolishing yielded suitable TEM specimens, but left the 600 grit scratches intact at the edge. The optical microscope could then be used to record the shape of the hole and orient the specimen with respect to the working direction. A picture of the hole was also taken on the TEM. Since relative rotations have been calibrated for the TEM, this allowed the working direction to be known for the TEM micrographs.

An example of a TEM/optical pair is shown in Fig. 5(a) and Fig. 5(b). By comparing the shape of the holes, the extruding direction (shown by the arrow in Fig. 5(a) may be located on the TEM micrograph. This information turned out to be critical in the interpretation of the results. In Figs. 6 and 7, grains of very high aspect ratio are visible (appearing dark). The associated diffraction patterns show that the long dimension of these grains corresponds to the  $<200>$  direction, and that the normal to the plan is  $<0\bar{1}1>$  . The large grain shown in Fig. 8 does not have so high an aspect ratio but the larger dimension still corresponds to the  $<200>$  direction. However, the plane normal in this case is  $<002>$  . In all cases, when the relative rotations were worked out, it was found that the working direction was parallel to the long axis of the grain and thus to the  $<200>$  direction. Similar grains were found in other samples cut in the same orientation. Figure 9 shows a bright field/dark field pair of an abnormal grain in a different specimen. Here the grain is aligned in the  $<200>$  direction, however, the plane normal does not correspond to any specific crystallographic direction. It is logical to make the conjecture that these elongated grains present in the as-hot worked condition grow into the coarse elongated grains upon annealing. Elsewhere in the specimen, the microstructure was like that shown in Fig. 10. This is a very fine micro-structure of grain size  $0.2 \mu\text{m}$  and with no preferred orientation.

Annealing at 1315°C (2400°F) for 10 minutes produced a grain size of approximately 2 mm by 0.6 mm, so that TEM specimens were essentially one single grain. As expected, the <200> direction of this grain was parallel to the extruding direction. This supports the theory that the abnormal elongated grains described above may be the embryos for the coarse grains. A bright field/dark field TEM pair is shown in Fig. 11. Note the extensive twins in the dark field picture. Twinning is so extensive that roughly half the structure is a mirror image of the other.

C. The Effects of Grain Shape on Nabarro-Herring and Coble Creep Processes  
(W. D. Nix)

It is well known that the high temperature creep properties of some alloys depend sensitively on both grain size and grain shape. Perhaps the most dramatic example of this was provided by Wilcox and Clauer [3-4] who showed that the creep strength of oxide dispersion strengthened metals increases markedly with both grain size and grain aspect ratio. At ordinary temperatures and stresses creep occurs by dislocation motion and grain boundary sliding, and the effects of grain shape must be explained in terms of these mechanisms. However, at very high temperatures and low stresses, below the threshold stress for dislocation creep [5], diffusional creep is expected to dominate. Whittenberger has provided direct evidence of diffusional creep on TD-Ni-20Cr by showing that particle free zones form at transverse grain boundaries [6]. In view of the possible importance of creep by diffusional flow in alloys having large grain aspect ratios, it seems worthwhile to study the effect of grain shape on diffusion creep.

In the present work we analyze Nabarro-Herring creep (lattice diffusion controlled) and Coble creep (grain boundary diffusion controlled) for the case of three dimensional grains shaped like rectangular parallelepipeds and packed together in the manner of an orthorhombic lattice. The results allow one to predict the diffusional creep rate for grains of any length, width and thickness. The limiting cases of grains with square cross-sections and two dimensional grains are also studied.

The grain geometry to be considered is shown in Fig. 12. For reasons of symmetry, it is necessary to consider only one octant of a grain having a length,  $L$ , width,  $W$ , and thickness,  $H$ . The exposed faces of the parallelepiped

represent the three adjoining grain boundaries. For three dimensional grains we define the grain size,  $S$ , as  $S = (LWH)^{1/3}$  and use two grain aspect ratios:  $R_1 = L/H$ ,  $R_2 = L/W$  to define the shape.

#### Lattice Diffusion Control

An exact solution for the diffusion equation has been obtained for the geometry shown in Fig. 12 and for the steady state flow boundary conditions. In the steady state, vacancies are uniformly removed from the transverse grain boundary and uniformly deposited on the longitudinal boundaries. Also, the distribution of tractions on the boundaries must be consistent with the applied stress state (mechanical equilibrium). The general solution to this problem in terms of the grain sizes  $S$  and the grain aspect ratios  $R_1$  and  $R_2$  is

$$\dot{\epsilon}/\dot{\epsilon}_0 = \frac{1}{(R_1 R_2)^{2/3}} \left\{ \frac{R_1^2 + R_2^2}{1 + R_1^2 + R_2^2} \right\} \quad (10)$$

where  $\dot{\epsilon}_0 = \frac{12 D_s \sigma \Omega}{L^2 k T}$  is the usual formula for Herring-Nabarro creep.

It is of interest to consider the special case in which the grains have a square cross-section. In this case  $R_1 = R_2 = R$  and Eq. (10) reduces to

$$\dot{\epsilon}/\dot{\epsilon}_0 = \frac{1}{R^{4/3}} \left\{ \frac{2 R^2}{1 + 2 R^2} \right\} \quad (11)$$

Notice that  $\dot{\epsilon}/\dot{\epsilon}_0 \sim R^{-4/3}$  as  $R \rightarrow \infty$ . Thus the diffusional creep rate decreases for large grain aspect ratios as expected. It is also interesting to note that  $\dot{\epsilon}/\dot{\epsilon}_0 \sim R^{2/3}$  as  $R \rightarrow 0$ . This indicates that the diffusional creep rate should also be low for very small grain aspect ratios. This would correspond to testing ODS alloys in the short transverse direction. These properties are shown in Fig. 13.

It is also possible to derive a special formula for two dimensional grains from the general solution. For the case in which  $W \rightarrow \infty$ , the result is

$$\dot{\epsilon}/\dot{\epsilon}_0 = \frac{1}{R_1 + \frac{1}{R_1}} , \quad (12)$$

where  $S$  in  $\dot{\epsilon}_0$  is now defined for two dimensional grains to be  $S = \sqrt{LH}$ .

For large grain aspect ratios  $\dot{\epsilon}/\dot{\epsilon}_0 \sim R_1^{-1}$  as reported by Raj and Ashby [7] but for very small aspect ratios  $\dot{\epsilon}/\dot{\epsilon}_0 \sim R_1$ . These results are shown in Fig. 14.

#### Grain Boundary Diffusion Control

The case of diffusional creep controlled by grain boundary diffusion has also been solved. Here the boundary conditions for the geometry shown in Fig. 12 are the same as discussed above but the governing diffusion equations are different. Diffusion equations are written for each of the three grain boundaries. They allow for vacancy diffusion in the plane of the boundary subject to the condition that vacancies are either uniformly removed or deposited on the boundary. A general solution to this problem leads to

$$\dot{\epsilon}/\dot{\epsilon}_{\infty} = \frac{\left(\frac{1}{R_1} + \frac{1}{R_2}\right) \left\{ \frac{R_2}{R_1} + \frac{R_1}{R_2} \right\}}{\left\{ \frac{1}{R_1} + R_1 + \frac{1}{R_2} + R_2 + \frac{R_2}{R_1} + \frac{R_1}{R_2} \right\}} \quad (13)$$

where  $\dot{\epsilon}_{\infty} = \frac{12 \delta D_{gb} \sigma \Omega}{k T S^3}$  is the usual formula for Coble creep.

For the case of grains having a square cross-section  $R_1 = R_2 = R$ , this reduces to

$$\dot{\epsilon}/\dot{\epsilon}_0 = \frac{2}{1 + R + R^2} . \quad (14)$$

Notice that for this case  $\dot{\epsilon}/\dot{\epsilon}_{\infty} \sim R^{-2}$  for large values of  $R$ . However, for small grain aspect ratios  $\dot{\epsilon}/\dot{\epsilon}_{\infty}$  approaches a constant value of 2. This behavior is shown in Fig. 13.

The two dimensional result can also be found by letting  $W \rightarrow \infty$ .

It is

$$\dot{\epsilon}/\dot{\epsilon}_{\infty} = \frac{1}{\sqrt{R} + 1/\sqrt{R}} \quad (15)$$

where again we have used the two dimensional grain size  $S = \sqrt{LH}$  in  $\dot{\epsilon}_{\infty}$ . Here for large  $R$ ,  $\dot{\epsilon}/\dot{\epsilon}_{\infty} \sim R^{-1/2}$  as reported by Raj and Ashby [7], but for small  $R$ ,  $\dot{\epsilon}/\dot{\epsilon}_{\infty} \sim R^{1/2}$ . These properties are shown in Fig. 14.

Throughout this work we have treated the Nabarro-Herring and Coble creep processes as separate and independent. It is common practice to express the total diffusional creep rate as the sum of these two terms. As discussed by Spingarn and Nix [8], this procedure is not strictly valid since the two solutions cannot be satisfied simultaneously. To underscore this point, we have chosen not to combine the two results in this work.

REFERENCES

1. J. Friedel, Dislocations, Addison-Wesley, Pub., (1964) p. 391.
2. J. P. Hirth and J. Lothe, Theory of Dislocations, McGraw-Hill, Pub., (1968) p. 465.
3. B. A. Wilcox and A. H. Clauer, Acta Met., 1972, 20, 743.
4. B. A. Wilcox and A. H. Clauer, "The Superalloys", C. T. Sims and W. C. Hagel, Eds., New York, 1972, John Wiley, Pub., p. 197.
5. R. W. Lund and W. D. Nix, Acta Met., 1976, 24, 469.
6. J. D. Whittenberger, Met. Trans. A, 1973, 4A, 1475
7. R. Raj and M. F. Ashby, Met. Trans. A, 1971, 2A, 1113.
8. J. R. Spingarn and W. D. Nix, Acta Met., 1978, 26, 1389.

IV. PUBLICATIONS, REPORTS AND DISSERTATIONS RELATING TO THIS AND  
PREVIOUS AFOSR GRANTS ON OXIDE DISPERSION STRENGTHENED METALS

A. Publications

1. J. H. Holbrook and W. D. Nix, "Edge Dislocation Climb over Non-Deformable Circular Inclusions", *Metall. Trans.*, 5, 1033 (1974).
2. M. Vikram Rao and W. D. Nix, "Creep in Binary Solid Solutions: A Possible Explanation for Anomalous Behavior", *Scripta Met.*, 7, 1255 (1973).
3. M. A. Burke and W. D. Nix, "Plastic Instabilities in Tension Creep", *Acta Met.*, 23, 793 (1975).
4. R. W. Lund and W. D. Nix, "On High Creep Activation Energies for Dispersion Strengthened Metals", *Metall. Trans.*, 6A, 1329 (1975).
5. R. W. Lund and W. D. Nix, "High Temperature Creep of Ni-20Cr-2ThO<sub>2</sub> Single Crystals", *Acta Met.*, 24, 469 (1976).
6. G. M. Pharr and W. D. Nix, "A Comparison of the Orowan Stress with the Threshold Stress for Creep for Ni-20Cr-2ThO<sub>2</sub> Single Crystals", *Scripta Met.*, 10, 1007 (1976).
7. J. H. Hausselt and W. D. Nix, "Dislocation Structure of Ni-20Cr-2ThO<sub>2</sub> After High Temperature Deformation", *Acta Met.*, 25, 595 (1977).
8. J. H. Hausselt and W. D. Nix, "A Model for High Temperature Deformation of Dispersion Strengthened Metals Based on Substructural Observations in Ni-20Cr-2ThO<sub>2</sub>", *Acta Met.*, 25, 1491 (1977).
9. R. F. Singer, W. Blum and W. D. Nix, "The Influence of Second Phase Particles on the Free Dislocation Density During Creep of Stainless Steel", *Scripta Met.*, 14, 755 (1980).
10. R. F. Singer, W. C. Oliver and W. D. Nix, "Identification of Dispersoid Phases Created in Aluminum During Mechanical Alloying", *Metall. Trans.*, 11A, 1895 (1980).
11. P. S. Gilman and W. D. Nix, "The Structure and Properties of Al-Al<sub>2</sub>O<sub>3</sub> Alloys Produced by Mechanical Alloying: Powder Processing and Resultant Powder Structure", (to appear in *Met. Trans.*).
12. W. D. Oliver and W. D. Nix, "The Effects of Strain Hardening in the Hydrostatic Extrusion of Axisymmetric Bi-Metal Rods", (to appear in *Metals Technology*).

B. Ph.D. Dissertations

1. R. W. Lund, "A Study of High Temperature Creep of Dispersion Strengthened Ni and Ni-20Cr", Ph.D. Dissertation, Stanford University (1975).
2. J. H. Holbrook, "A Theoretical Investigation of Creep of Dispersion Strengthened Crystals", Ph.D. Dissertation, Stanford University (1976).
3. P. S. Gilman, "The Development of Aluminum-Aluminum Oxide Alloys by Mechanical Alloying", Ph.D. Dissertation, Stanford University (1979).

C. Oral Presentations (Speaker Underlined)

1. W. D. Nix, "On the Existence of Steady State Creep", Department of Mechanical Engineering, University of Colorado, Boulder, Colorado (December, 1973).
2. W. D. Nix, "Plastic Instabilities in Tension Creep", Air Force Conference on: Fracture and Fatigue of Two Phase Materials - Effects of Plastic Instability, Fairborn, Ohio (September, 1974).
3. W. D. Nix, "High Temperature Creep of Dispersion Hardened Single Crystals", TMS-AIME (invited paper), University of Toronto (May, 1975).
4. W. D. Nix, "When are Back (Threshold) Stresses Meaningful?", 1977 Gordon Conference on Physical Metallurgy, Holderness School, Plymouth, N.H., June, 1977. (Invited paper).
5. P. S. Gilman and W. D. Nix, "Mechanical Alloying of Al-Al<sub>2</sub>O<sub>3</sub> Alloys, presented at 1978 Spring Meeting of AIME in Denver.
6. W. C. Oliver, R. F. Singer and W. D. Nix, "Formation and Identification of Particles in Dispersion Strengthened Aluminum", presented at 1980 Spring Meeting of AIME in Las Vegas.
7. W. C. Oliver, "Mechanical Alloying", presented at Department of Materials Science and Engineering, Industrial Affiliates Meeting at Stanford, June, 1980.
8. W. D. Nix, "Creep of Dispersion Strengthened Metals", presented at Exxon Research Laboratory, Linden, New Jersey, February, 1980.

V. PROFESSIONAL PERSONNEL

The following personnel of the Department of Materials Science and Engineering at Stanford have been engaged in this research program:

Principal Investigator:

Dr. William D. Nix, Professor

Co-Principal Investigator:

Dr. Robert Sinclair, Assistant Professor

Graduate Research Assistants:

Mr. Warren C. Oliver, B.S., University of Tennessee  
M.S., Stanford University

Mr. Russell R. Mueller, B.S., Elmhurst College

Ms Jean K. Gregory B.S., Massachusetts Institute of  
Technology

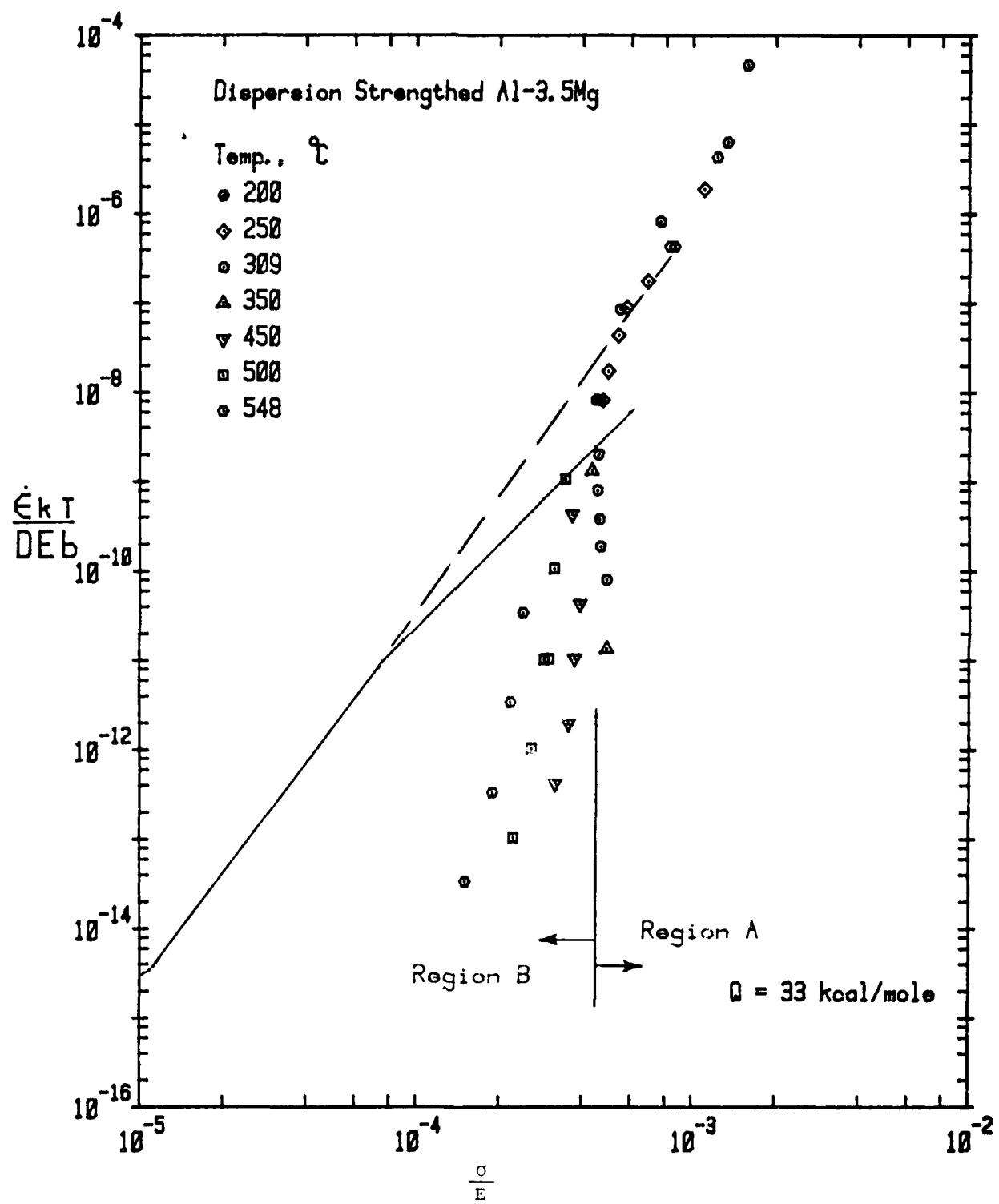


Figure 1. The temperature compensated strain rate versus the modulus compensated stress. The line is for Al + 3.5% Mg containing no oxide dispersion. The data points are for IN 9051, a dispersion strengthened Al + 3.5% Mg.

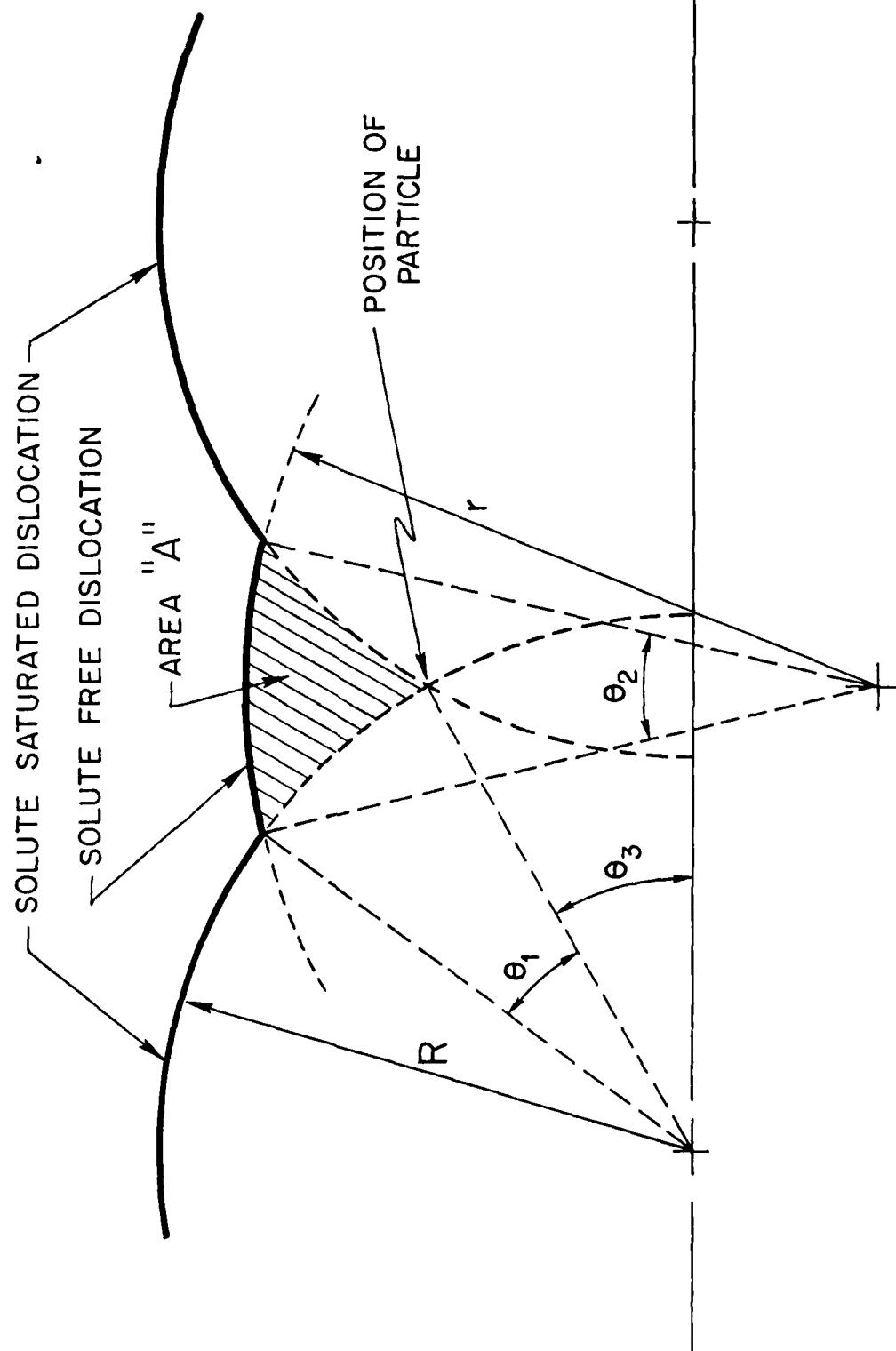


Figure 2. The geometry of a dislocation line that has just passed a particle at point A.

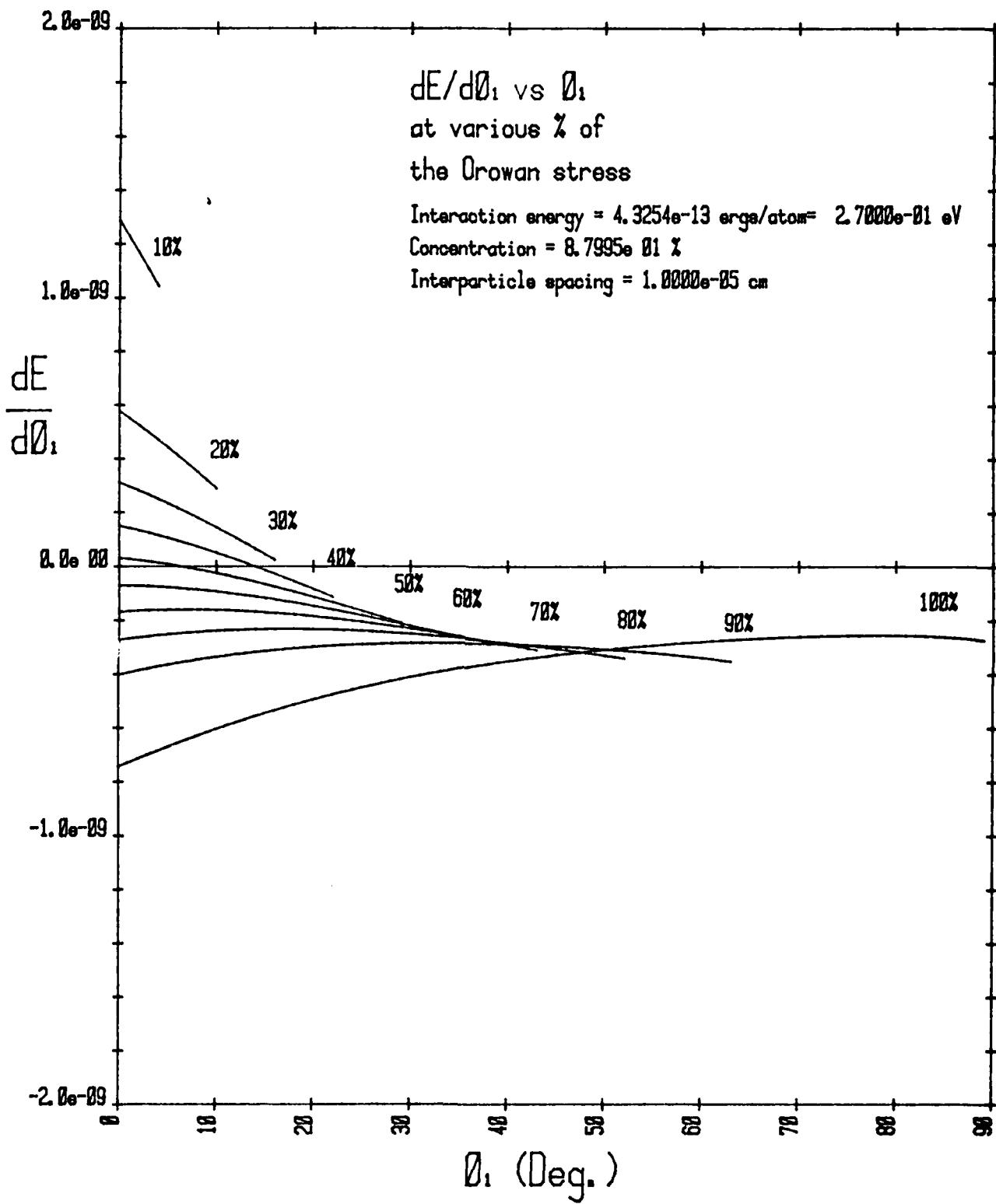


Figure 3. The variation of energy with respect to  $\theta$ , at various fractions of the Orowan stress.

Stress for Break Away (% of Orowan)  
vs Temperature (Deg. C)

Interparticle spacing =  $1.0000e-05$  cm  
 $\gamma_0 = 2.7000e-01$  ev =  $4.3254e-13$  ergs/atom

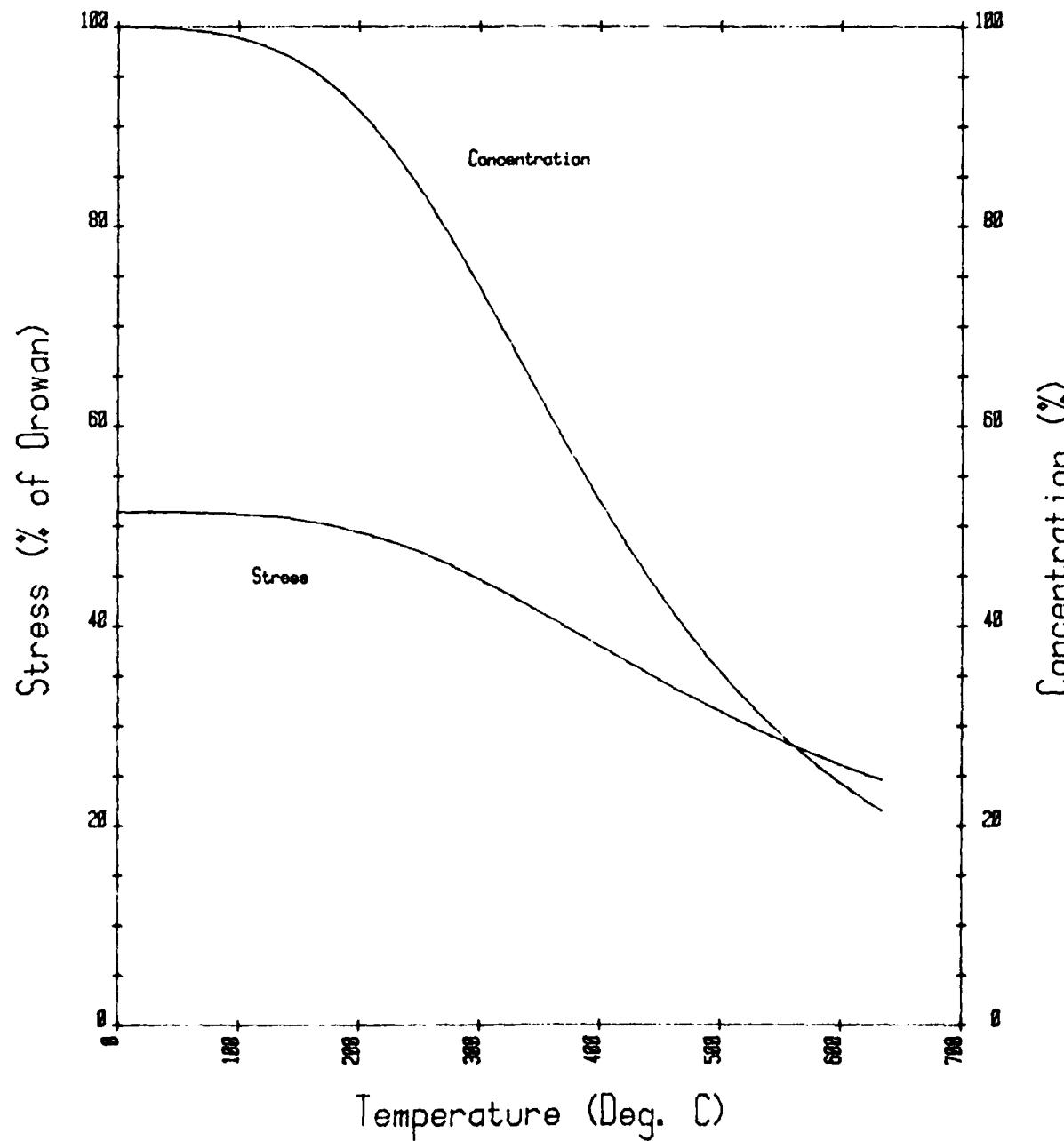


Figure 4. The minimum stress to un-zip a dislocation from a solute atmosphere and the solute concentration at the core of the dislocation as a function of temperature.

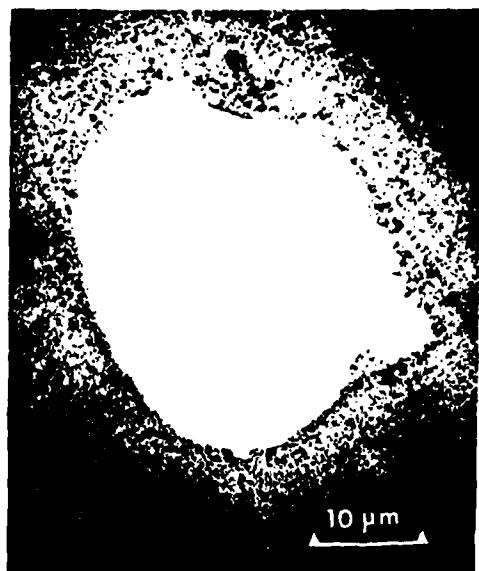
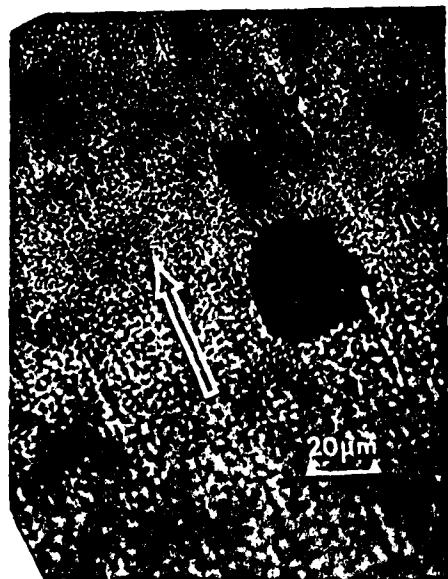


Figure 5. (a) Optical and (b) TEM micrographs of specimen hole. Extrusion direction is known for (a), (shown by the arrow) and inferred in (b) from the shape of the hole.

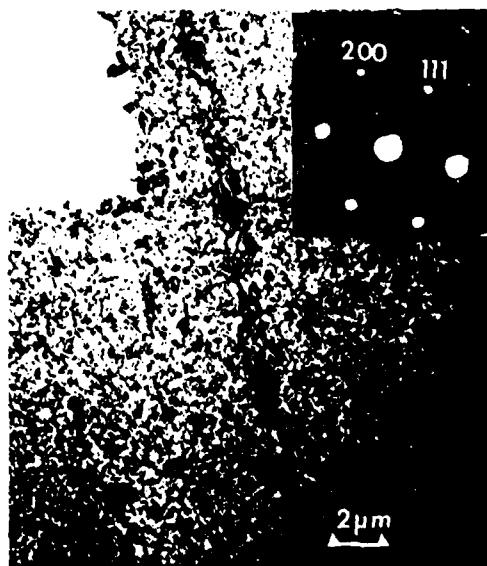


Figure 6. Abnormal grain in as hot-worked MA 753. Long direction of the grain is parallel to  $<200>$  is parallel to the working direction. Zone axis is  $<01\bar{1}>$ . 120 kV.



Figure 7. Different abnormal grain from the same specimen as Figure 6 . Long direction of the grain is parallel to  $<200>$  is parallel to the working direction. Zone axis is  $<01\bar{1}>$  . 120 kV.

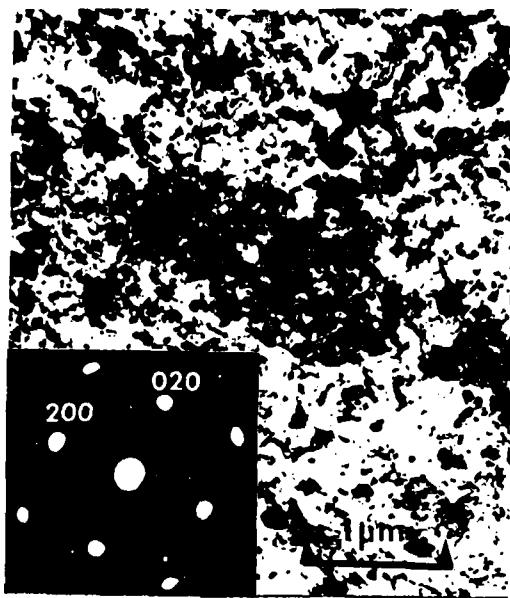


Figure 8. Abnormal grain in MA 753. Here the aspect ratio is not as high as Figures 6 and 7. Long direction of grain is parallel to the working direction. Zone axis is  $\langle 002 \rangle$ . 120 kV.

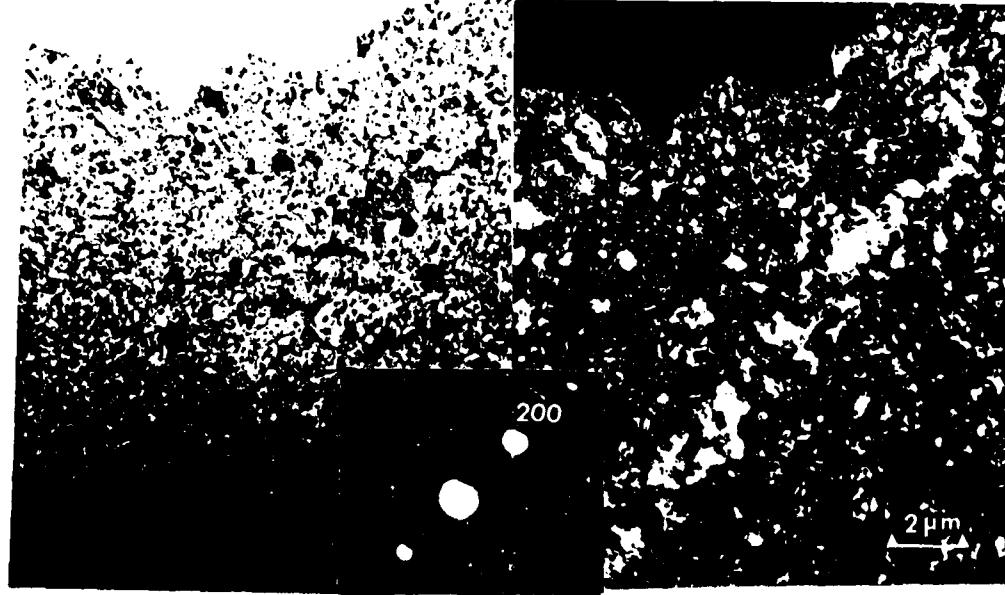


Figure 9. Bright field/dark field pair with diffraction pattern of abnormal grain. Again, long direction of grain is parallel to  $\langle 200 \rangle$  is parallel to the working direction. Zone axis not well-defined. Dark field obtained with (200) reflection. 120 kV.

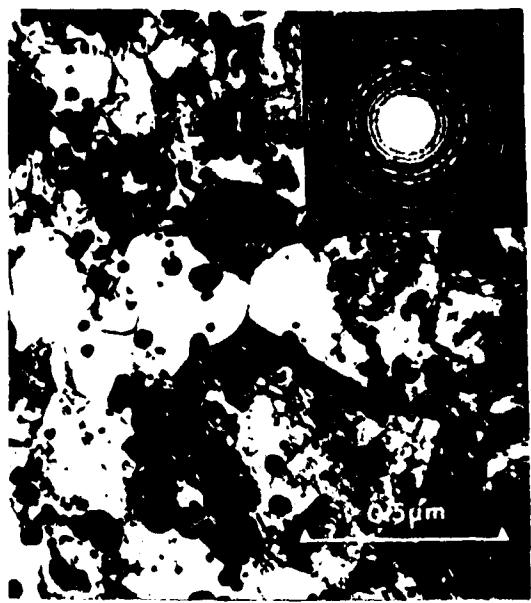


Figure 10. Bright field and ring diffraction pattern showing a typical microstructure of as hot-worked MA 753. Grains are roughly  $0.2 \mu\text{m}$ , equiaxed, and randomly oriented.



Figure 11. Bright field/dark field pair from annealed MA 753. Extensive annealing twins are present. Dark field obtained with  $\langle 200 \rangle$  reflection. 120 kV.

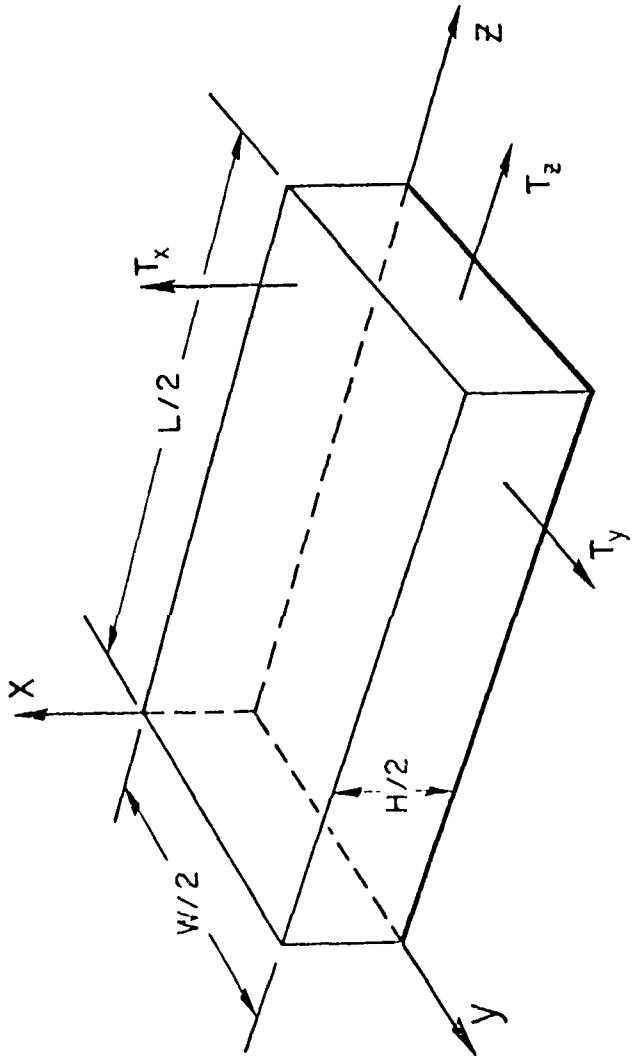


Figure 12. Illustration of one octant of a grain of arbitrary length, width and thickness.

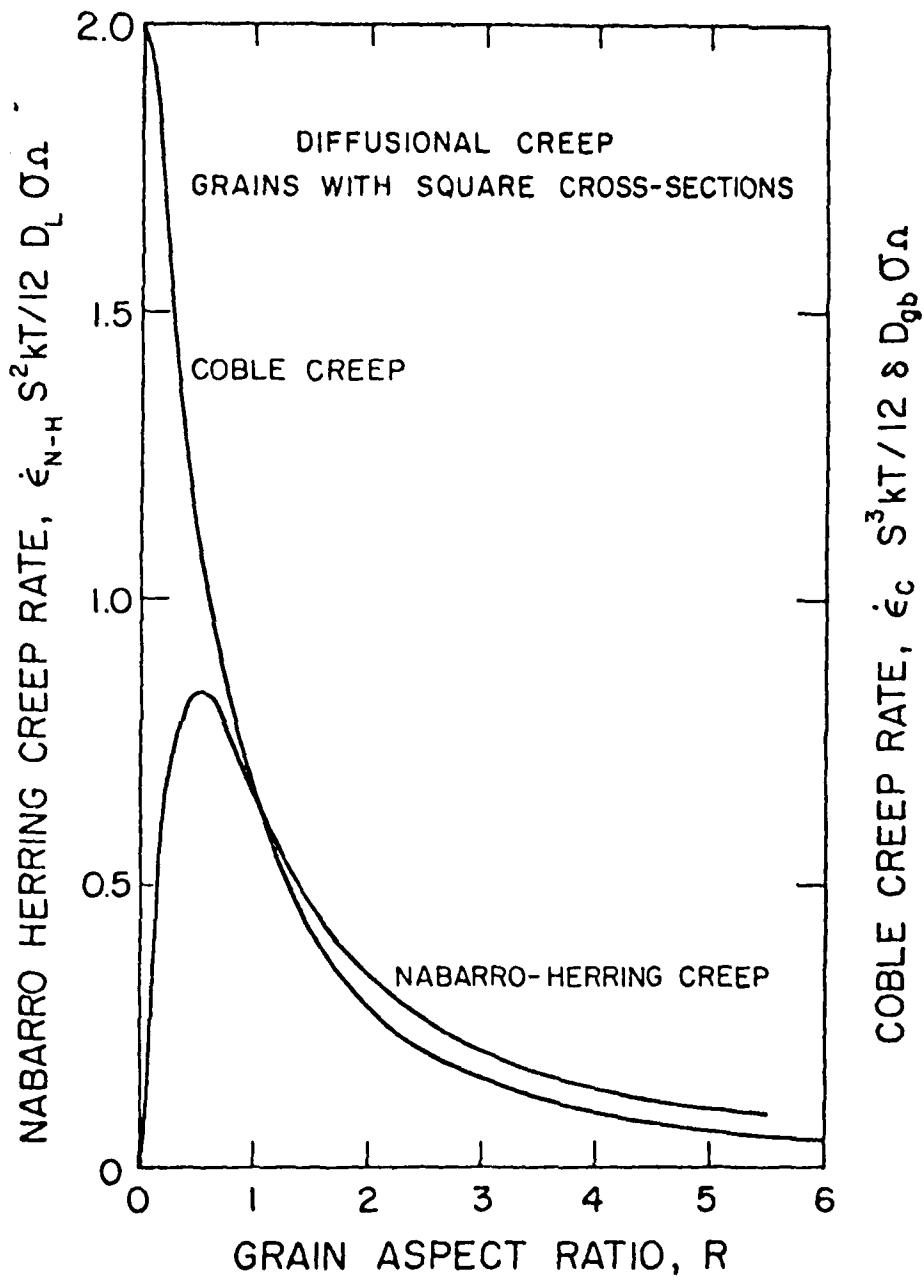


Figure 13. Effect of grain aspect ratio on Nabarro-Herring and Coble creep for grains with square cross-sections.

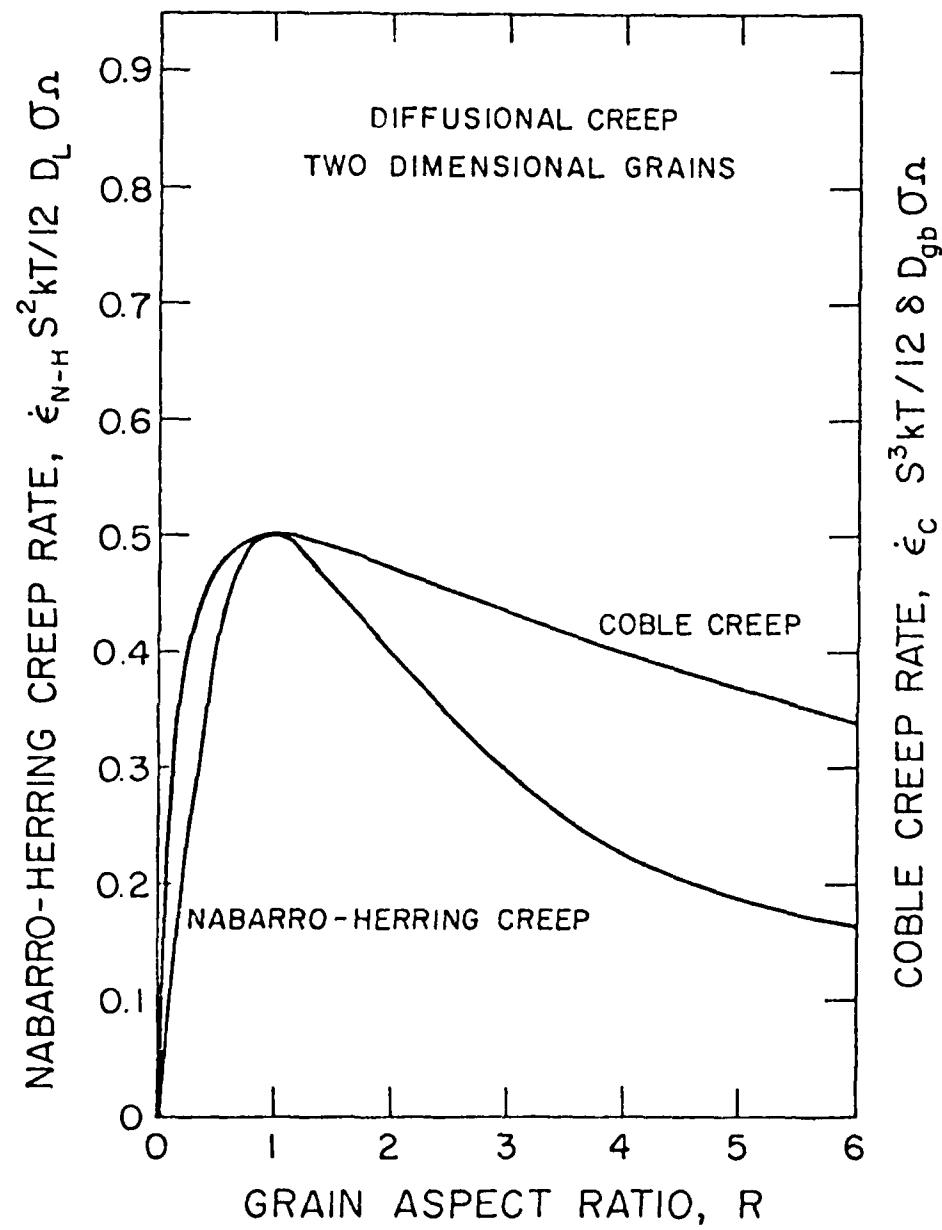


Figure 14. Effect of grain aspect ratio on Nabarro-Herring and Coble creep for two dimensional grains.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The purpose of this research is to develop a better understanding of the structure and properties of oxide dispersion strengthened metals. The main thrust of this work has been to understand the mechanical alloying process by which many ODS alloys are now made and to synthesize new structural materials with mechanical alloying. We are also interested in the mechanisms by which elongated grain structures form during the processing of ODS alloys. Some of our recent work has been directed toward the understanding of the high temperature mechanical properties of ODS aluminum alloys.		

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